

DEPARTMENT OF AGRICULTURE
DAIRY COMMISSIONER'S BRANCH
OTTAWA, CANADA

CHEMICAL INVESTIGATIONS RELATING TO DAIRYING
UNDERTAKEN IN 1904

BY

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DEPARTMENT OF AGRICULTURE
DAIRY COMMISSIONER'S BRANCH
OTTAWA, CANADA

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LETTER OF TRANSMITTAL.

OTTAWA, May 15, 1905.

To the Honourable

The Minister of Agriculture.

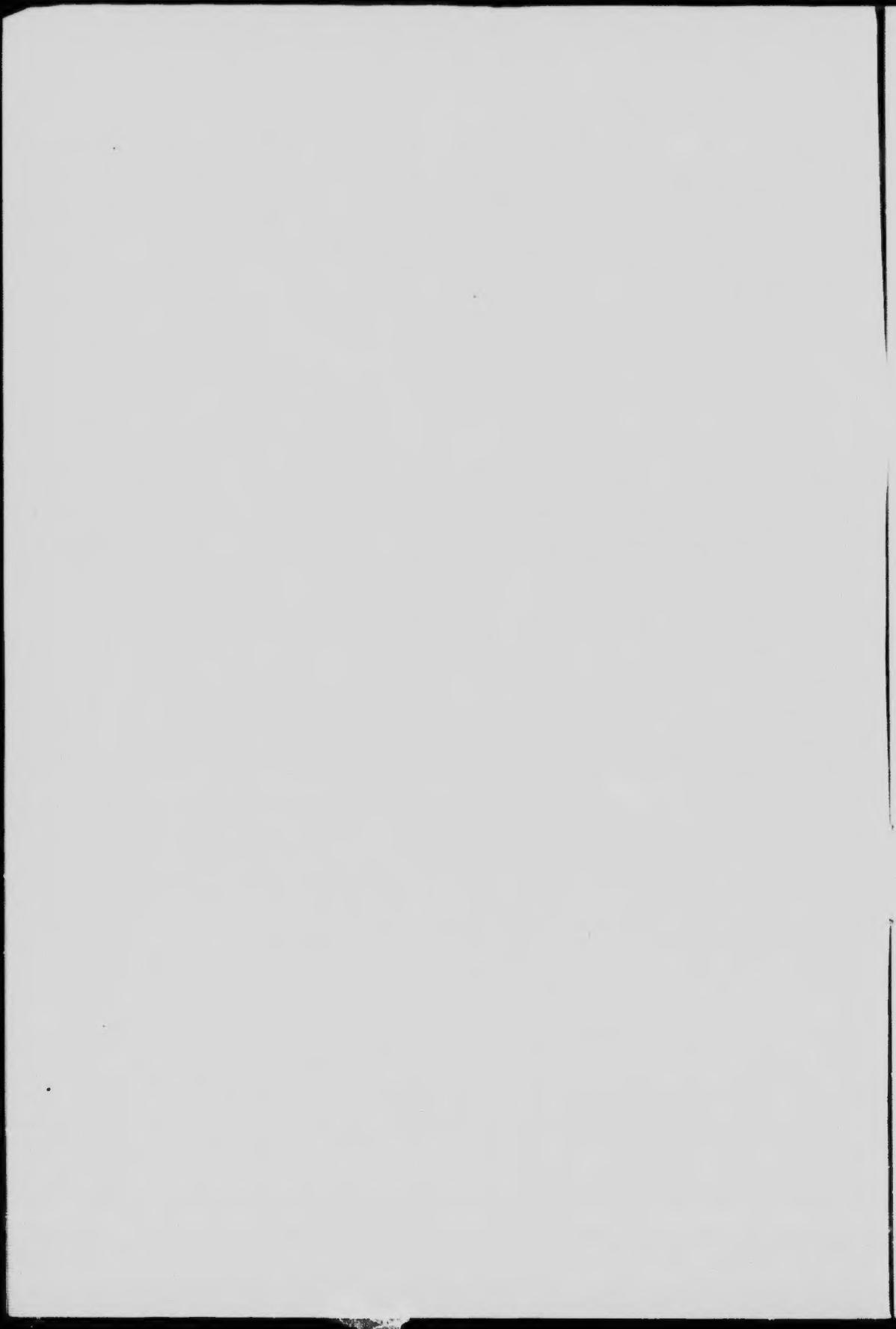
SIR,—I have the honour to present herewith Bulletin No. 6, Dairy Commissioner's Series, 'Chemical Investigations Relating to Dairying Undertaken during 1904,' by Mr. Frank T. Shutt, Chemist, Experimental Farms, and beg to recommend that it be printed for distribution.

I have the honour to be, sir,

Your obedient servant,

J. A. RUDDICK,

Dairy Commissioner.



CHEMICAL INVESTIGATIONS RELATING TO DAIRYING UNDERTAKEN DURING 1904.

BY FRANK T. SHUTT, M.A., F.I.C.,
Chemist, Dominion Experimental Farms,

From time to time investigations bearing on matters relating to the dairy interests of Canada are referred to the laboratories of the Experimental Farms by the Dairy Commissioner. In the present bulletin a concise report is given of the more important of these researches undertaken during the year 1904, under the following titles:—

The Examination of Milk preserved with Hydrogen Peroxide by the Process of F. G. Korch, Copenhagen, Denmark.

A Critical Study of the Buttermaking Process of James Estep.

The Analysis of a 'Milk Powder' obtained by the Evaporation of Whey.

The Volatile Acid Content of Fat from Two-Year-Old Cheese.

Recent Apparatus devised for the Determination of Water in Butter.

REPORT ON SAMPLES OF MILK PRESERVED BY HYDROGEN PEROXIDE, BY PROCESS OF F. G. KORCH, COPENHAGEN, DENMARK.

By this process 'the raw milk is heated to a temperature of about 50 degrees C., (122 degrees F.), and then a certain very small quantity of a solution of hydric peroxide is added, the milk stirred or shaken, and kept at said temperature for some hours.' It is stated by Dr. Korch that the milk is now free from hydric peroxide and perfectly sterile. Dr. Korch further says 'that if the milk is desired to be kept in a condensed state it can be evaporated as much as required in a vacuum apparatus and the hydric peroxide added.'

The samples here reported upon were those which had been submitted by the inventor of the process to the High Commissioner for Canada in London, England, by whom they were transmitted to the Department of Agriculture, Ottawa.

The box contained two large tins and three smaller tins. In each of the larger tins there was a bottle holding about 8 fluid ounces of milk; in the smaller tins the milk (about 10 ounces), was not bottled. All were securely sealed. One bottle and one of the smaller tins were forwarded, as received, to Dr. W. T. Connell, Queen's University, Kingston, for bacteriological examination.

Milk in Bottle.—This was quite fluid, and the cream which had risen was easily re-incorporated by slightly warming and gently agitating the milk. The milk had the appearance of having been diluted or skimmed, being well described as 'poor' or 'thin.'

It had a slight but distinct taste or flavour, rather unpleasant and not unlike that produced by the action of heat on milk. There was no marked or characteristic smell. It had a very slightly alkaline reaction.

Examination was made for the following preservatives: Hydrogen peroxide, ferrulin, salicylic acid, boracic acid, and borax. The first mentioned, hydrogen peroxide, was found to be present, but none of the others could be detected.

Analysis.

Specific gravity	1.033
Total solids	11.39
Fat	2.45
Ash71
Curd (nitrogen x 6.25)	3.00

The percentages of total solids and of fat are less than those in normal milk, and certainly much below the standards or limits recognized for average herd's milk. The indications of skimming are extremely strong. This milk would in Canada be accounted as below standard, since it is generally held that genuine herd's milk must contain at least 3·5 per cent fat.

Milk in Tin.—This was found to be partially churned, but not curdled. It was of a creamy colour and possessed a greasy or oily smell. Clinging to the inside of the cover and walls of the tin were a few lumps of black material which proved to be largely fat impregnated with iron rust. A deposit gradually settled on allowing the milk to stand.

The reaction was slightly alkaline.

The examination for preservatives proved the presence of hydrogen peroxide. Negative results were obtained for formalin, salicylic acid, borax and boracic acid.

Analysis.

Specific gravity	1·058
Total solids	25·71
Fat	8·08
Ash	1·44
Curd (nitrogen \times 6·25)	6·3

These data indicate a concentration of the milk to about one-half of its original volume.

Dr. W. T. Connell, who made a very careful and thorough bacteriological examination, states as the result of his research that both of these milks are sterile. The claims of the inventor in this respect are, therefore, correct. That the milk does not remain sterile for any length of time after opening the containing vessel is, however, also shown by Dr. Connell, whose report in full is attached hereto.

Judging from the samples submitted for examination, it is not possible to draw a favourable conclusion regarding the palatability of either of these milks or their suitability for general use.

KINGSTON, ONT., March 7, 1904.

MR. FRANK T. SHUTT, M.A., F.I.C.,
Chemist, Dominion Experimental Farms,
Ottawa.

DEAR SIR,—I have completed the examination of the two specimens of Danish preserved milk (Korch process), which you forwarded me on February 18th ultimo. I append results of my examination.

1.—Bottle Packed in Large Tin.

Bottle held about 8 ounces. The milk was perfectly fluid with cream well mixed. There was no acid odour, but the milk had an acrid bitter taste clinging with persistency to palate. The Babcock test showed this sample to contain 2·6 per cent fat. None of the ordinary preservatives were detected. The fat globules were very small on microscopic examination. Bacteriologically, the milk was sterile, giving no growth of bacteria, yeasts or moulds though tested on various media. Samples removed and placed in sterile tubes and kept some at 37 degrees C., and others at 20 degrees C., under both aerobic and anaerobic conditions, are still unchanged and free from bacterial growth (18 days). Bacteria introduced into this milk grew without difficulty.

2.—Small Can Holding About 10 Ounces.

Milk in this evidently condensed, the specific gravity being 1·076 and fat 3·8 per cent. The cream was not so well mixed, being present partially in the form of distinct small granules. This sample had a sweet, nutty flavour at first with a bitter, per-

sistent after taste which was very disagreeable. This sample contained traces of formaldehyde. Bacteriologically, this sample was sterile. Bacteria grow in it slowly. Evidently this milk was partially skimmed before condensing. Various persons tested both samples as to their palatability, and all agreed that the milk was decidedly unpalatable, in fact disagreeably so. I cannot see that such milk would stand any chance of being adopted for general use for this reason alone.

Yours truly,

(Sgd.) W. T. CONNELL.

A CRITICAL STUDY OF THE BUTTERMILK PROCESS OF JAMES ESTEP.

In the specification the inventor claims to have perfected 'a new and useful improvement in the art or process of making butter.' He states: 'This invention relates to the treating of cream or milk with the herein described substances whereby butter may be made direct from sweet cream, and has for its object the production of a greater quantity of butter in less time and with less labour; and the butter thus produced will be free from disagreeable flavour resulting from foul weeds, decayed roots, garlic, willows, &c., eaten by cows, and may, by the exercise of proper care, be kept indefinitely without spoiling.'

As regards the claim that a larger quantity of butter is obtained by this method, Mr. Estep states: 'I find that by using my process, and comparing it with the process in use at present, that I can produce, on the average, an extra half pound of butter to every gallon of cream, and that the time taken for the churning is only about one-half that ordinarily taken at present.'

Without entering into details it may suffice to say that this method consists in adding to the cream, either sweet or sour, a certain quantity of a mixture made up of pepsin, sugar of milk, alum, and salt-petre, the proportions of which are given.

A quantity of sweet cream in uniform composition was divided into three equal portions by weight. 'A' was allowed to ripen, and was churned in the usual manner. 'B' was allowed to ripen, the compound then added according to directions, and churned. To 'C,' while still sweet, the required quantity of the compound was added, and the cream immediately churned.

The temperature of the three churning was the same, viz., 62° F.; the times of churning were for 'A' 10 minutes, for 'B' 15 minutes, and for 'C' 18 minutes.

It is apparent, therefore, that instead of reducing the time required for churning this compound lengthened it.

The butters were salted in the granular state at the rate of $\frac{1}{2}$ oz. per lb., and immediately worked and weighed. The weights of butter severally obtained are as follows:

	Lbs. Ozs.
'A,' from ripened cream, ordinary method.	11 1
'B,' " " " Estep's process.	11 0
'C,' " 'sweet' " " "	11 1

These results show very clearly that the claim of an increased yield of butter by the Estep process is unfounded. It is evident that the use of the compound does not effect the weight of the butter produced. According to the claim made, from the quantity of cream used in this test, there should have been from 'B' and 'C' about 2 pounds more butter than from 'A.'

Analysis of Butters.

	'A'	'B'	'C'
Water	15'75	14'21	14'64
Fat	80'83	82'41	81'49
Curd	1'53	1'36	1'43
Salt	1'89	2'02	2'44

The above data do not indicate any significant differences in composition; the butters are such as might be obtained from any three churnings by the usual procedure.

To ascertain if any of the mixture added remains in the butter, the ash of 'B' and 'C,' which it may be remarked were only normal in amount, were analytically examined. The results were negative, showing that these chemicals had all been washed out with the buttermilk.

It is also of interest to notice that the proportion of curd in the butter from the treated cream is normal.

Dairy experts tested these butters while still fresh and after a period of three months, and reported no marked influence on the flavour or keeping qualities by the treatment. The butter from the sweet cream, however, did not keep so well as that from the ripened cream.

In conclusion, we might say that this investigation furnished no support to the claims that this process increases the yield of butter or reduces the time of churning.

MILK POWDER FROM THE EVAPORATION OF WHEY.

This material is produced simply by the evaporation of whey and may consequently be considered a by-product in the manufacture of cheese. The patentee, Mr. Daniel Ramsum, states that a similar product is made and largely used in several countries of northern Europe, and that it is considered both palatable and nutritious. It is further stated by Mr. Ramsum that when made into a paste it can be used as a substitute for butter and cheese, and that it may also form the basis of an acceptable beverage.

As received, this product was in the form of a light yellow powder, with a slightly sweetish taste and rather pleasant odour. No taint was observed after six months' preservation in a stoppered bottle. On analysis the following data were obtained:—

Composition of Milk Powder.

Moisture	3'73
Albumin and nitrogenous bodies	12'81
Fat	3'91
Milk sugar (by difference)	72'27
Ash	7'28
	100'00

We may notice first the small percentage of water present. To this may be largely attributed its apparently excellent keeping qualities.

In flesh-forming compounds (albumin), this powder approximates certain classes of cheese, though considerably poorer in this respect than Canadian cheese, which usually contains in the neighbourhood of 35 per cent proteids.

In the manufacture of cheese the incorporation of the fat is usually very complete. We should not, therefore, expect to find a large percentage of this constituent in this

by-product. Nevertheless, when considered as an article of food this milk powder is found to contain a very fair proportion of fat.

Practically three-fourths of the product is milk sugar, a soluble and easily digested carbohydrate. The percentage of this constituent in normal milk is about 475.

The mineral matter or ash is about ten times the amount present in milk, and naturally consists largely of phosphates of lime and potash.

Summing up, we may say that the analytical data show clearly that this preparation has a distinct nutritive value. As to the economy of the process and the possibility of such a material becoming popular as an article of food in Canada, the writer, however, has his doubts.

THE VOLATILE ACID CONTENT OF FAT FROM TWO-YEAR-OLD CHEESE.

In a recent case before the courts in England in which the genuineness of certain Canadian cheese was questioned, the point was raised as to the changes which might take place in the composition of the fat on keeping the cheese in cold storage for a considerable period, say as for one or two years. It was held by some that such changes, if any, might result in a decrease of the volatile fatty acid content, and thus furnish data similar to those obtained when cheese has been adulterated with foreign fat.

To obtain data upon this important matter we have analysed a cheese made at Woodstock in September, 1902, and subsequently kept by the Dairy Commissioner for 26 months in cold storage. Though somewhat strong, this cheese on its arrival at the laboratory was found to be sound and good. Its composition, as revealed by analysis, is as follows:—

Water.....	29'62
Fat.....	36'55
Curd.....	32'86
Salt.....	'97
	100'00

These figures do not indicate any abnormality as regards composition, but may be regarded rather as representing the quality of the average output of Canadian cheese factories.

In the determination of the proportion of the volatile fatty acids, more commonly known as the Reichert Number, the fat was dissolved out of the cheese by means of cold ether. The ether was then allowed to spontaneously evaporate and the fat submitted to analysis according to one of the most approved methods.

The Reichert Number is obtained from the distillation of the saponified fat, after acidification, by the titration of the distillate with a deci-normal soda solution. For genuine butter fat this number, according to Reichert, Hehner, Allen and other authorities, lies between 12'5 and 15. When butter and cheese are adulterated with foreign fat, as with margarine, this number is very much lower. The closely concordant results of duplicate analyses of the fat of this two-year-old cheese gave an average Reichert Number of 13'45. This gives most satisfactory proof that in this case at least cold storage for a period of more than two years had not materially affected the volatile fatty acid content.

Further experiments are now in course with cheese, the complete analysis of which has been made, to ascertain possible changes on keeping under varying conditions of temperature, &c., for lengthy periods. It is satisfactory, however, to note that this investigation, so far as it has been prosecuted, has furnished data supporting the contention that age and cold storage do not affect genuine cheese so as to render it liable to be confounded with the 'filled' or adulterated article.

APPARATUS FOR THE DETERMINATION OF WATER IN BUTTER.

CARROLL'S TESTER.*

This apparatus comprises a special measure for the butter, glass tubes in which the butter is melted and the separated water measured, a water-bath furnished with a spirit lamp and a wooden rack to hold the tubes when the reading is made.

The butter being pushed out of the measure into the tube, the latter is corked and placed upright in the water-bath. The tubes are then allowed to remain (with occasional removal and agitation), in the boiling water for about 45 minutes, by which time it is stated that all the water will have separated from the fat and collected in the lower and graduated portion of the tube.

A large number of trials were made, following the directions as closely as possible, but satisfactory results could not be obtained. In the first place, it was found practically impossible in a large proportion of the tests to make out with any degree of sharpness the junction of the watery layer and the separated butter fat, owing to the presence of suspended curd. Secondly, the readings in every instance were much too low, even when made on the supposition that the line of demarkation between the water and the fat was above the curd.

The following data include the results obtained by this tester on two samples of butter. For the purposes of comparison the percentages of moisture in the same butters obtained by gravimetric analysis are given:—

Moisture.

	By Gravimetric Analysis.	By Carroll's Tester.
Sample 'A,' print butter, C.E.F.	13'76	$\begin{cases} 9'0 \\ 8'0 \end{cases}$
Sample 'B,' print butter, C.E.F.	13'13	$\begin{cases} 5'0 \\ 6'0 \\ 4'0 \end{cases}$

It is evident from these results that in our hands the apparatus has not proved trustworthy, and that we could not recommend it even when only approximate percentages are required.

THE GELDARD BUTTER TESTER.**

By this method the water content of the butter is found by determining the loss by weight that follows when the butter is submitted to such a temperature that will result in the evaporation of the water, but not cause decomposition of the fat. Fifty grains of butter are weighed into a small porcelain dish together with a metal stirrer and a small quantity of an attenuating material. The dish is now placed on the 'evaporator' and the gas burner lit. Very shortly after the butter melts, bubbles of steam are seen to rise and escape. The elimination of the water is assisted by occasionally stirring the melted butter. In '8 to 15 minutes' the evaporation of the water will be complete (known by the cessation of the formation of froth), and the dish is removed from the evaporator to cool and be re-weighed. The difference between the first and second weighings, multiplied by 2, gives the percentage of water in the butter. It will be seen from this outline of the operation that the method is practically one of gravimetric analysis.

* The Dairy Supply Co., Ltd., London, W.C.

** G. R. Geldard, 34 Stanley Road, Manchester, England.

All the necessary apparatus to conduct the test, including the balance and weights, gas burner, dish, stirrer, &c., is furnished by the patentee, and the accompanying instructions are explicit and readily followed.

The degree of accuracy obtainable by this process was ascertained by determining the percentage of water in two samples of butter, and making estimations by an approved method of gravimetric analysis for comparison. The following data were obtained:—

	<i>Moisture.</i>	<i>By Analysis.</i>	<i>By Geldard's Apparatus.</i>
Sample 'A,' print butter, C.E.F.	13'76		{ 13'6 13'8 13'6
Sample 'B,' print butter, C.E.F.	13'13		{ 13'2 13'2 13'2

These results are extremely satisfactory, and show that the method is capable of furnishing data in close accordance with those obtained by accepted methods of analysis. It is, of course, necessary that the weighings be made with accuracy, and that throughout the operation great care should be exercised to avoid any loss save from evaporation of the water of the butter.